



# TECHNICAL NOTE

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## CERAMIC VACUUM ULTRAVIOLET ION CHAMBERS

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by

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### SUMMARY

A new ceramic ion chamber for making vacuum ultraviolet radiation measurements in the laboratory and outside the earth's atmosphere has been developed with the cooperation of the Coors Porcelain Company, Golden, Colorado, the manufacturers.

This detector has excellent electrical insulating properties and a high work function. It is practically impervious to the toxic effects of gases. An improved window-sealing technique aids in prolonging the life span of this ion chamber.

This paper gives a detailed description of the fabrication, gas filling, and sealing techniques. The ion chamber calibration procedure is briefly outlined; a forthcoming paper will cover this subject in detail.

The usefulness of the chamber in the 1050-1500A spectral region is noteworthy because the detector can be made to operate over a relatively narrow bandpass with a sharp cutoff (rejection of photons) on either side of the selected wavelength region. Other useful properties include the high quantum yield, the extremely low noise level, and the capability to serve as a gas-gain multiplier.



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## INTRODUCTION

The ion chamber technique is a simple, reliable method of measuring vacuum ultraviolet radiation both in the laboratory and outside the earth's atmosphere. Since photon counters of earlier design frequently remain operational only over short-term rocket investigations, it was necessary to improve the reliability of these devices for space-probe and satellite applications.

Ion chambers and photon counters suitable for space science programs were first developed and flown by the U. S. Naval Research Laboratory in the 1950's (References 1 and 2). Previously developed ion chambers were fabricated from materials such as copper shells, kovar and kovar-glass seals, silver chloride window seals, etc.; but these proved inferior because of cracking seals and electrical leakage and also because the copper and glass used were photoemissive surfaces. As a result, ion chambers were efficient only for several months and the time interval between fabrication and flight had to be held to a minimum.

The Coors Porcelain Company, Golden, Colorado,† beginning early in February, 1959, assisted in the development of a ceramic ion chamber shell having excellent electrical insulating properties, a multi-layer inner cathode surface with a high work function, and materials practically impervious to the toxic effects of gases. Along with the improved ion chamber shell, a new window-sealing technique was developed which increased the life span of the detector from several months to more than a year. This paper will describe in detail the fabrication techniques used. The three basic steps in fabrication are:

1. Assembly — The assembly of the ceramic-metal envelope-flange and the optical window into a vacuum-tight unit.

\*Report submitted November 22, 1961.

†NASA, Space Sciences Division, Ion Chamber, Model 1-B, Drawing No. B7-011B.

2. Gas Filling — The gas distillation process and the back filling of the ion chamber.
3. Calibration — The measurement of the quantum efficiency or photo-ionization yield of a ceramic ion chamber.

## ASSEMBLY

The impervious ceramic-metal envelope may be purchased as a prefabricated unit consisting of a high density alumina shell, into which is brazed a kovar anode pin, guard-ring-connector housing, and a gas filling tube. The inner circumferential surface (cathode) is a multi-layer surface (Figure 1). The optical windows of barium fluoride, calcium fluoride and lithium fluoride discs\* are carefully mounted on the ceramic-metal envelope assembly by means of a stamped silver flange (0.005" sheet) that has been gold plated (0.002" thick).

The steps to be followed in assembling the ion chamber are listed in the outline which follows:

### A. Cleaning of envelope and flange

First check the inner shell for loose metal and sharp surfaces and check the filling tube for fractures and obstructions. Then immerse the envelopes and flanges in an ethyl alcohol (200 proof) bath and ultrasonically clean for five minutes.

\*Harshaw Chemical Co., Cleveland, Ohio.

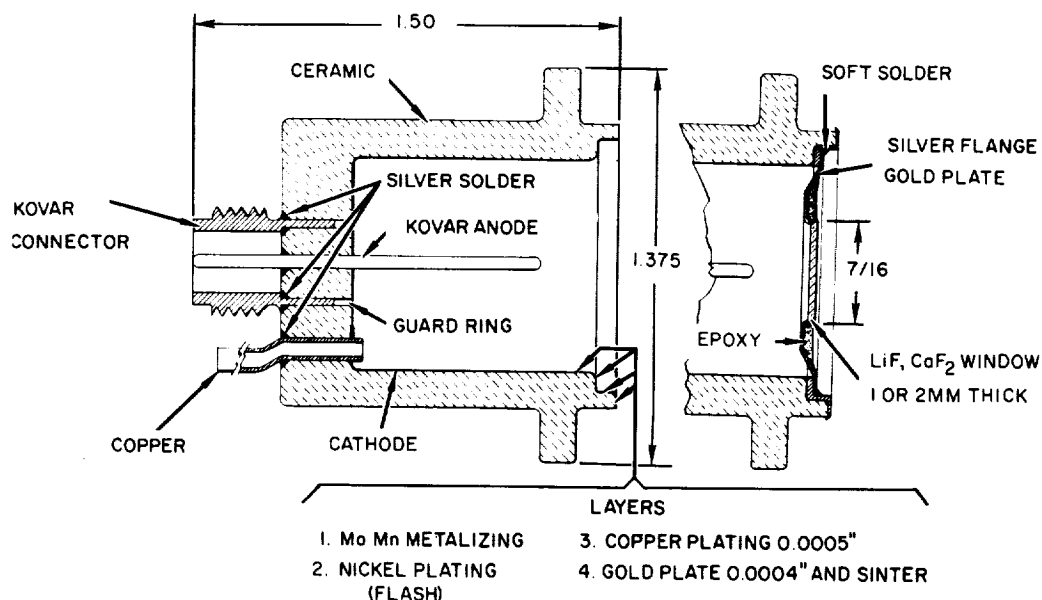


Figure 1 - Diagram of the NASA ion chamber



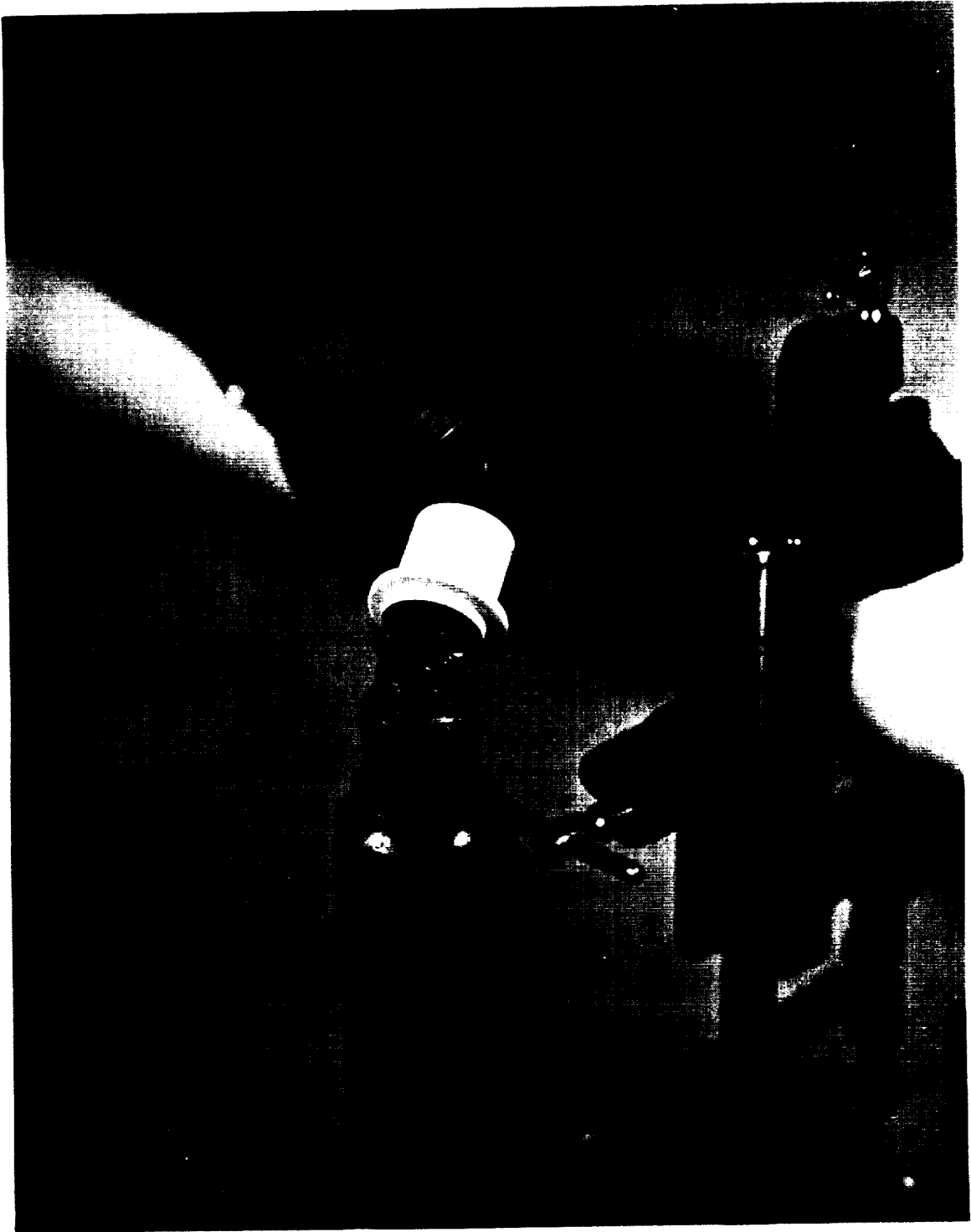


Figure 2 - Soldering the flange

**B. Drying**

Remove from the alcohol bath, shake off the excess alcohol and dry the shell rapidly with a hot air gun.

**C. Fluxing and tinning**

1. Mount the soldering iron (with modified tip) in an upright position (Figure 2).
2. Heat, clean, flux,\* and re-tin soldering iron.
3. Preheat the ion chamber shells to about 200°F on a hot plate, or in an oven, before tinning the flange. This is important since the flange retaining rim is easily cracked by applying too much pressure or by prolonged, rapid heating.
4. Apply flux to the ion chamber flange and mate it to the soldering iron ridge. Keeping contact as brief as possible, rotate the chamber with a slight clockwise, counterclockwise motion until the chamber flange is evenly and completely tinned on the flange seat and on the side walls.

**D. Soldering flange to envelope**

1. Remove the collar from the flange (Figures 3 and 4) with scissors, cutting back to the proper diameter of the ion chamber mating flange. The flange is pressed from a 0.005" pure silver sheet and, in the process, an extra, unnecessary lip or rim is formed. In fact, the amount of pressure required to seat the flange, as pressed, usually cracks the flange lip or produces uneven seating.
2. Flux only the silver flange seating rim and place it in the ion chamber mating flange, using no more pressure than necessary to hold the silver flange in place.
3. Mate the envelope and flange to a hot iron and solder them into position quickly, using only enough pressure to seat the flange evenly.
4. Then place the ion chamber in the rotating, motor-driven device shown in Figure 4. Place the preset soldering iron against the rotating ion chamber and apply flux and solder, making a uniform fillet around the periphery of the pre-metallized envelope and flange.

**E. Removal of flux and contaminants**

1. When the chamber being made has cooled to 140°F, rinse the soldered envelope-flange assembly inside and outside in running hot water.
2. Ultrasonically clean the assembly in an ethyl alcohol bath for five minutes.
3. Heat the ion chamber shell-flange with a hot air gun until dry.
4. Rewash the ion chamber in a second and separate alcohol bath for 5 minutes and repeat the drying process.
5. Check the anode pin for center and examine the window-flange for smoothness.

\*All-State Duzall (160).

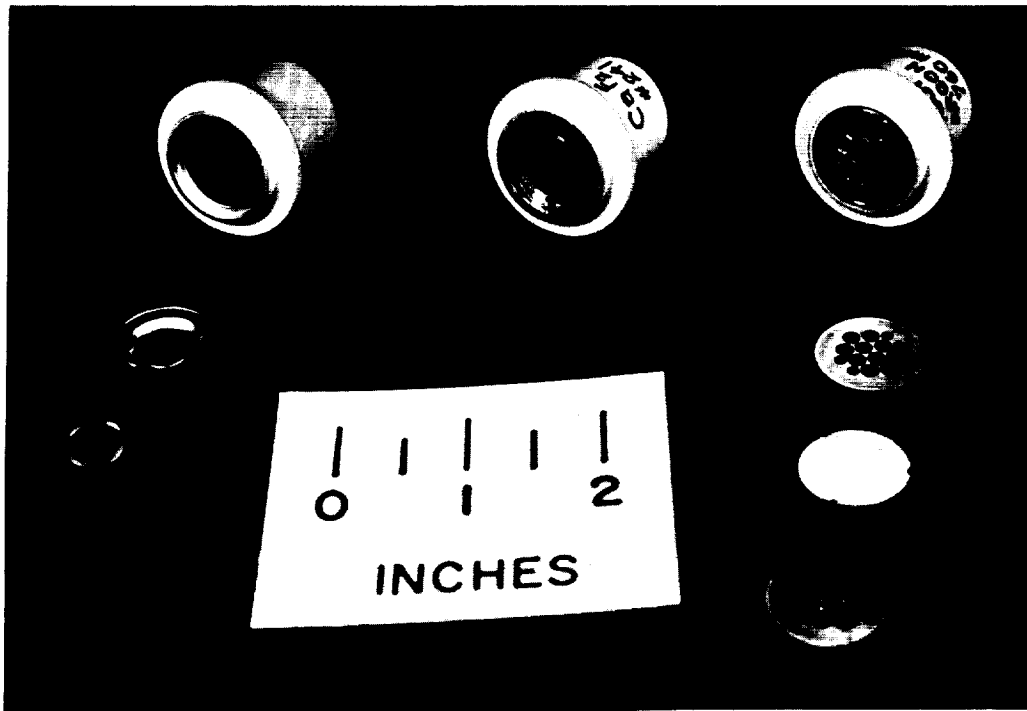


Figure 3 - Window and flange of the ion chamber

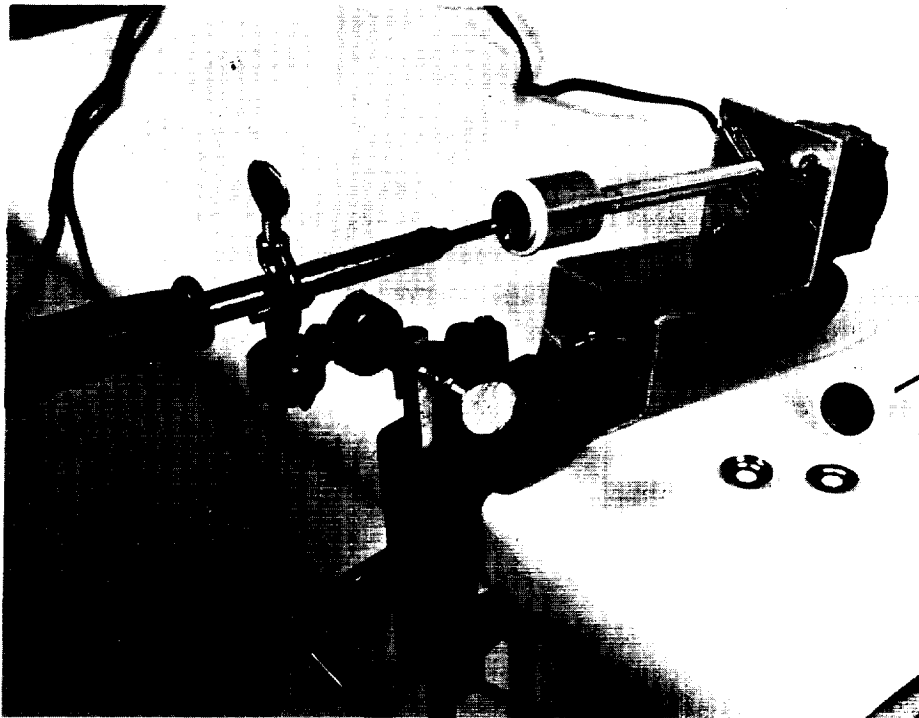


Figure 4 - Final soldering seal of the flange

6. From this point to the completion of the fabrication of the ion chambers, dessicate the assemblies after each operation is completed.

F. Preparation of epoxy resin sealant

This epoxy resin was chosen from a number of sealants tested because of its plastic properties, when cured, and because it does not appear to be affected by the various filling gases.

1. The epoxy resin\* employed consists of two parts — Hysol 12-007A and 12-007B; they should be kept under refrigeration. This resin should not be used for window-sealing purposes for a period longer than recommended by the manufacturer.
2. The mixture is composed as follows: Hysol 12-007A epoxy (formerly Hysol 6233A), 100 parts by weight; Hysol 12-007B catalyst or hardener (formerly Hysol 6233B), 80 parts by weight. Caution should be exercised when measuring the parts. Use a separate cup and mixing stick for each constituent and discard them after use.
3. Mix the parts by pouring part B into part A using a paper cup, which is not plastic coated, as the mixing container. Do not mix more than 1/3 of a container per mix (explanation is in section G-2).
4. The mixture has a pot life of about four hours, but should not be used after three hours for window-sealing purposes.
5. Stir the mixture thoroughly. Avoid whipping the epoxy mixture since this causes an excessive amount of air bubbles.

G. Evacuation of bubbles from the mixture

1. Place the mix in a bell jar and evacuate the jar, taking care that the mixture does not overflow.
2. Overflowing is prevented by letting air into the vacuum system through a needle valve at appropriate moments. Repeat the evacuation and admission of air many times. The degassing rate may be observed by watching the increased size of the bubbles. When outgassing passes through a critical point the surface of the foam level diminishes very rapidly and the admission of air should be terminated. Then pump the mix for several minutes until the surface shows only an occasional air bubble.
3. Bring the mixture to atmospheric pressure and, following this, avoid excessive probing of the epoxy.

H. Selecting and cleaning the fluoride windows

1. Measure window material of  $\text{LiF}$ ,  $\text{CaF}_2$ , and  $\text{BaF}_2$  for transmission properties; they should exhibit high transmittances in the desired wavelength region prior to installation. Select only windows which are free of heavy cleavage planes, cracks, chipped edges, and interface multiplicity.

\*Hysol Corp.

2. The windows should be desiccated or in a vacuum; they should not be touched with bare fingers. Wear latex gloves or finger cots while selecting and cleaning the windows.
3. Hold the clean windows with plastic-tipped tweezers and clean them by immersion in pure ethyl alcohol; then rub them with a clean Kimwipe or lint-free lens tissue. Repeat this step if necessary.

I. Sealing on the fluoride windows

1. Letter and number all the ion chambers with porcelain ink and record them in a log book. This log should contain a complete history of each ion chamber.
2. Apply a small bead of epoxy around the window sealing lip with a tapered stick (see Figure 1). This requires only enough epoxy to seal the window to the flange. Avoid or work out any air bubbles which may appear.
3. With plastic-tipped tweezers, drop the window in the exact center of the flange, avoiding overflow or creeping of epoxy on the window. Work out any air bubbles which appear. Adequate wetting for sealing occurs in the early stages of curing since heating causes the epoxy to flow and wet, although not excessively.

J. Curing of epoxy

1. Preheat the air-vacuum oven to 90°-100°C.
2. Place ion chambers in special trays, insert them into the oven, and cure them for two hours on the first coat.

K. Second epoxy coat — fillet around window

1. Remove the ion chambers from the oven. While still hot apply more epoxy (what is left over after application of the first coat is still usable) to form a fillet between the window and flange.
2. Cure the second coat at 90°-100°C for 1-1/2 hours.

L. Third epoxy coat — over entire flange

1. Before removing ion chambers for the third window coat, prepare a smaller amount of fresh epoxy containing: Hysol 12-007A — 6-1/4 parts by weight; Hysol 12-007B — 5 parts by weight.
2. With a camel's-hair brush paint a final coat of epoxy on the window-flange, covering the solder seals and the ceramic-to-metal seal as well.
3. Cure the third epoxy coat at 90°-100°C for 1-1/2 hours.
4. When the third coat has cured, remove the ion chambers from the oven. While they are still hot, invert each ion chamber and paint the copper filling tube from the porcelain to 1/8" above the top of the connector, around the base of the kovar to the porcelain seal. Using a fine wire or a hypodermic needle coat the inside of the

connector base and around the anode pin at the porcelain-metal seal. This insures vacuum tightness and eliminates electrical leakage. Paint a thin film of epoxy over the inked ion chamber number and the window material designation on the side of the ion chamber.

5. Cure the final epoxy coating at 90°-100°C for 1-1/2 hours.

M. Test for plasticity of epoxy

1. Although the hysol epoxy mixture usually cures properly, check each mix after curing. If the mixing ratios are correct the ion chamber window seals cured in the oven will be hard. The epoxy seal should not crack when a sharp stick (cotton swab applicator) is pressed against the surface.
2. Make a second check by allowing the unused portion of each mix to cure overnight at room temperature. Remove the sample from the paper cup. The cured sample should be dry and plastic.

N. Leak-testing ion chamber seals and windows prior to gas filling or storage

1. Seal the ion chambers to the ten-port manifold vacuum system as shown in Figure 5 via the gas filling tube which is placed in an "O" ring quick-coupling. Then evacuate the chambers to a pressure of  $1 \times 10^{-6}$  mm Hg and conduct a helium leak test.
2. Heat the ion chambers to about 100°C with a hot air gun, and again conduct a helium leak test. If a tube passes the helium leak test while hot, but indicates an excessive pressure after being isolated from the vacuum system momentarily, the tube is contaminated and should be removed from the group.
3. The recommended filling pressure is  $1.4 \times 10^{-6}$  mm Hg or lower.
4. Unfilled or filled ion chambers should be stored in desiccators except for filling or calibration.
5. Avoid touching the windows with fingers and avoid exposing the crystals to moisture or other contaminants.

O. Recording ion chamber history

A complete record should be kept of each chamber as follows: (1) tube number; (2) type window; (3) leak test; (4) gas filling, pressure and date; (5) type gas; (6) end use; (7) remarks.

P. Reclaiming leaking, inferior, or dead ion chambers

1. Saw open inferior or dead ion chamber filling tubes with a jewelers' hack saw. Care should be taken to saw only until a small opening occurs, which will allow the chamber to reach atmospheric pressure slowly, without pulling loose metal particles into the chamber.
2. If the tube is long enough, leak-detect and refill.

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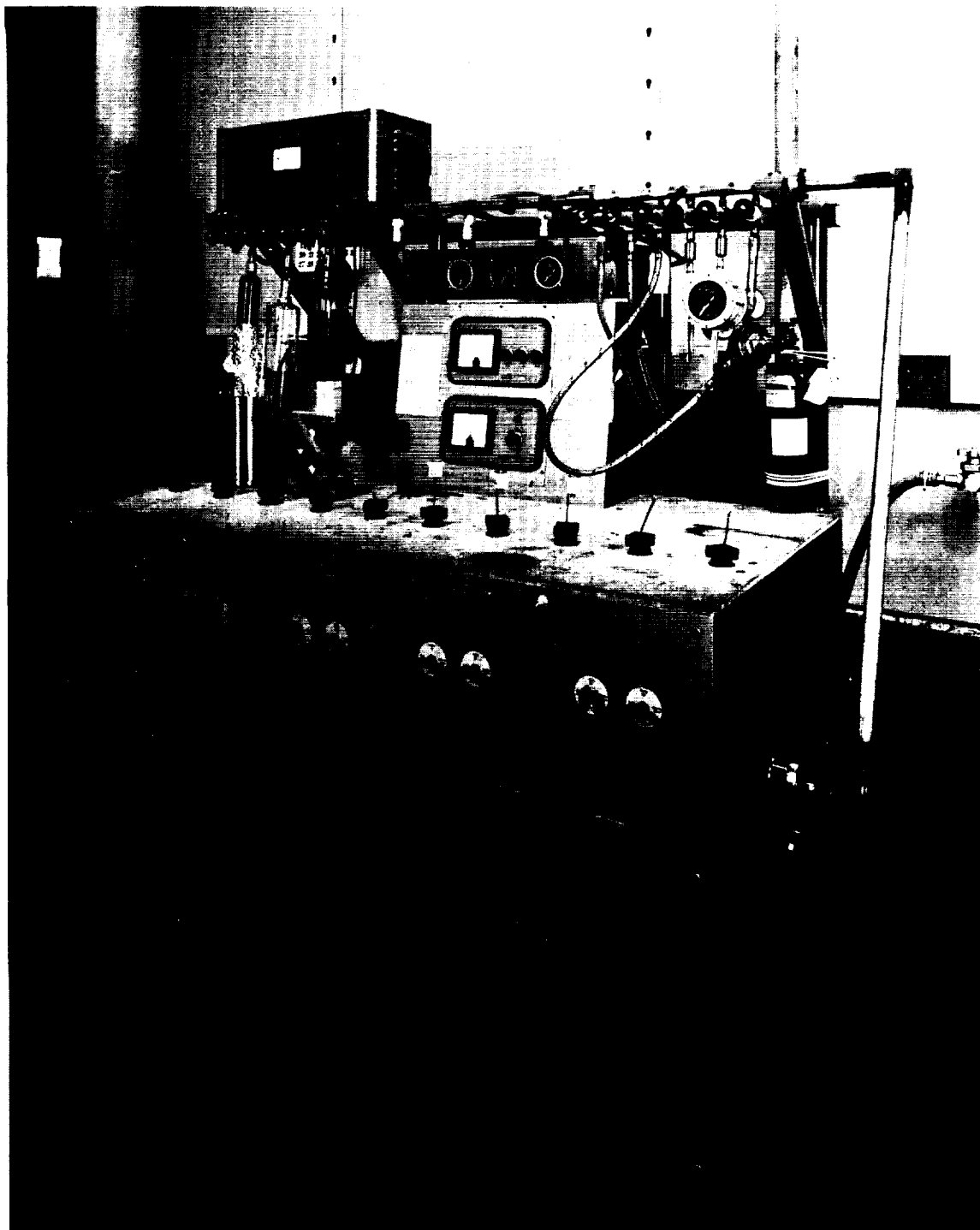


Figure 5 - Ten-port manifold

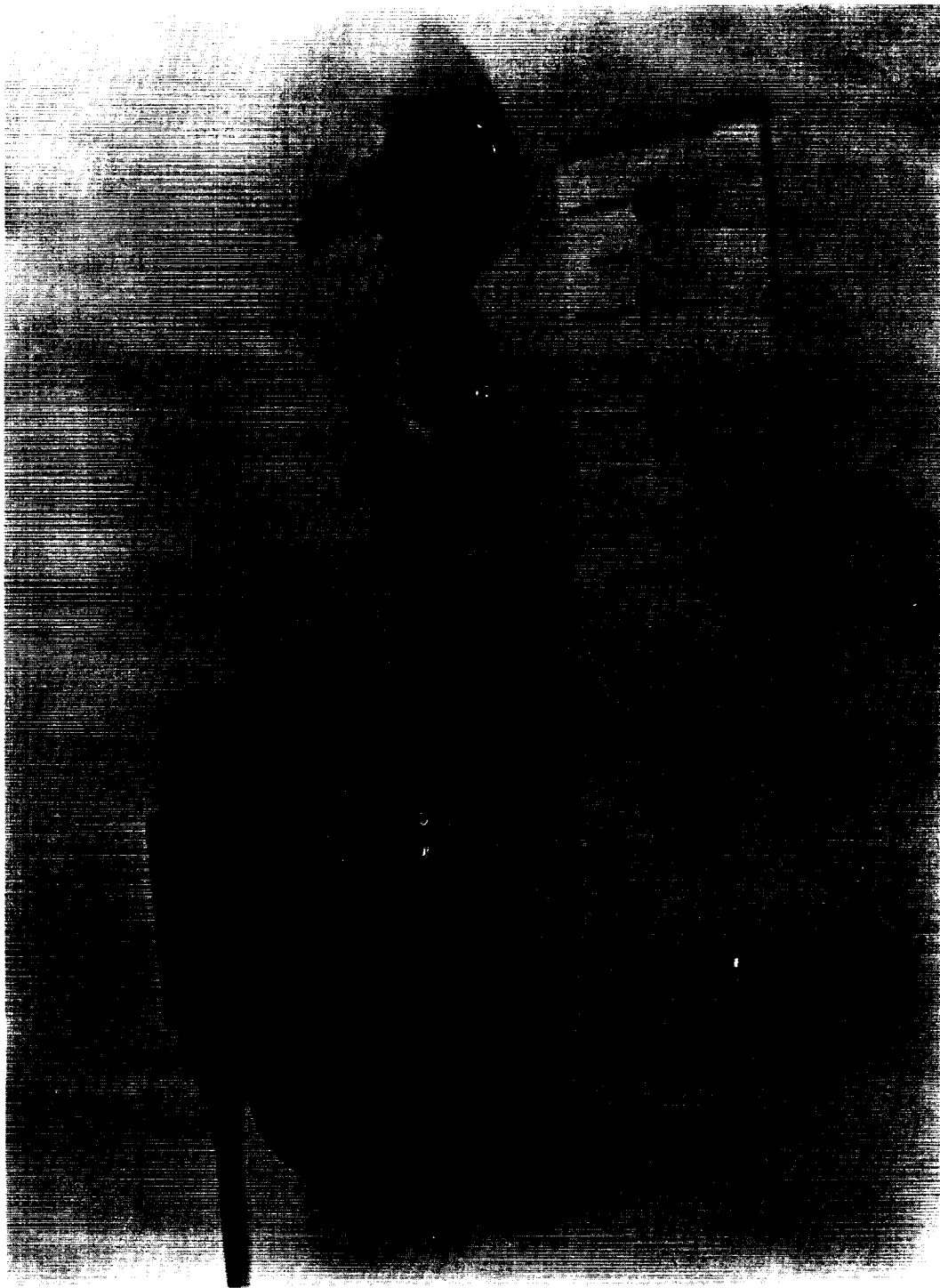


Figure 6 - Other tubes



3. For ion chambers with cracked windows, or extremely short filling tubes, a complete disassembly is required.
  - a. Immerse the ion chambers in a beaker of epoxy solvent\* until the window falls free and the epoxy peels. This soaking process usually takes 24 hours.
  - b. Remove the ion chambers from the solvent and air dry; then ultrasonically wash them in an alcohol bath.
  - c. Remove the flanges by heating with the soldering iron.
  - d. In the case of a short fill tube, silver-solder a tube extension with a larger diameter over the remaining section.
  - e. If the gold plating is defective or spot peeled, a second copper and gold plating usually is all that is required. This requires the removal of the gold and copper plating down to the nickel coating.
4. Now reprocess the reconditioned ion chamber as a new unit.

**Q. Other tube fabrication**

Figure 6 shows a variety of tubes which undergo the same fabrication techniques as described for a ceramic ion chamber. They are, left rear then left to right in front:

1. A Geiger-Muller counter with a 0.015" beryllium window epoxy-sealed directly to the chamber flange. The fill is 7 mm ethyl formate and 603 mm xenon;
2. A wide-angle copper ion chamber with a 2 mm LiF window epoxy-sealed directly to the chamber flange. The fill is 20 mm nitric oxide;
3. A soft X-ray ion chamber with a 0.005" beryllium window, copper-plated edge, soft soldered, epoxy-sealed. The fill is 775 mm xenon;
4. A soft X-ray ion chamber with a 0.003" aluminum window, epoxy-laminated between two brass support plates. The wafer is epoxy-sealed to the chamber flange. The fill is 760 mm argon;
5. A ceramic ion chamber with a 2 mm LiF window silver flange soldered to the chamber flange, window epoxy-sealed. The fill is 15 mm carbon disulfide;
6. A copper ion chamber having a 2 mm LiF window silver flange soldered to the chamber flange, window epoxy-sealed. The fill is 20 mm nitric oxide (Reference 3).

## **GAS FILLING SYSTEM**

The gas filling equipment (Figure 5) used for filling ion chambers, proportional counters, etc., consists of three independent systems connected to a central port manifold. Each of the first nine ports may be opened independently to a rough vacuum (atmospheric

\*Ram De Solv 292, Ram Chemical Inc.

pressure to 25 microns), a high vacuum (25 microns to  $10^{-7}$  mm Hg), and a filling system (0 absolute to 800 mm Hg). The tenth port is used to evacuate the filling system and make distillations. The process used in gas filling an ion chamber with nitric oxide follows:

#### A. Distillation of the filling gas

Although commercially obtained nitric oxide gas\* (Reference 4) is normally 99.0 percent pure, further distillation is required if the gas is to be used as an ionization medium. The impurities to be removed are nitrogen ( $N_2$ ), oxygen ( $O_2$ ), nitrogen dioxide ( $NO_2$ ), nitrous oxide ( $N_2O$ ), nitrous anhydride ( $N_2O_3$ ), nitrogen tetroxide ( $N_2O_4$ ), nitrogen pentoxide ( $N_2O_5$ ), and water ( $H_2O$ ). In order to distill:

1. Immerse a lecture bottle of nitric oxide gas, connected to the gas filling manifold, in a bath of dry ice and alcohol. This cold trap freezes out  $NO_2$  (at  $-9.3^\circ C$ ),  $N_2O_5$  (at  $+30^\circ C$ ), and  $H_2O$  (at  $0^\circ C$ ).
2. Evacuate the gas distillation manifold to a pressure of  $1 \times 10^{-6}$  mm Hg or less.
3. Pressurize the distillation system or manifold to 760 mm Hg (atmospheric pressure) with NO gas from the lecture bottle. Open a second flask, cooled to liquid nitrogen temperature, to the manifold. The NO will solidify.
4. Then close the flask valve and the trapped NO will be isolated from the manifold.
5. Since the absolute pressure gauge does not return to zero, a noncondensable gas (probably  $N_2$  and  $O_2$ ) is evidently present which must be pumped off. Again open the valve on the flask to the evacuated manifold, and then close it. A rise in manifold pressure will be noted. Now evacuate the gas filling manifold to  $1 \times 10^{-6}$  mm Hg pressure. Repeat this process until there is little or no apparent change in pressure when the NO flask is opened to the system.
6. Remove the liquid nitrogen trap and replace it with the alcohol and dry ice trap on the flask containing solid NO.
7. The solid NO quickly begins to sublime ( $-151.8^\circ C$ ); the valve is then opened to the manifold. Admit the purified gas to a storage bottle and valve-off.
8. Remove the alcohol and dry ice bath from the NO trap. Two different solids are noticeable in the trap, one yellow, the other blue. This combination is generally believed to be a mixture of the oxides of nitrogen, but the exact composition of the residue is not known.
9. Evacuate the entire gas filling system to  $1 \times 10^{-6}$  mm Hg pressure.

#### B. Gas filling an ion chamber

The ion chamber is filled with the proper gas to a specified pressure:

1. Seal the ion chamber on the main port with an "O" ring-flange and evacuate to  $1 \times 10^{-6}$  mm Hg or less.

\*Matheson Co.

2. Heat the ion chamber to about 200°C continuing to evacuate until it cools to room temperature.
3. Isolate the ion chamber from the vacuum system and switch it to the gas filling manifold by closing the vacuum valve and opening the gas fill valve.
4. Admit nitric oxide from the storage flask into the manifold and ion chamber to an absolute pressure of 20 mm Hg. (For other gases, distillation processes and filling pressures are available.)
5. It appears that while several ion chambers on this system are filled simultaneously, the quantum efficiencies are most nearly similar from tube to tube if each tube (in consecutive order) is valved off from the gas filling manifold and pinched off before being separated from the system for soldering. The other method of valving off, pinching off, and soldering the first chamber, then proceeding to the second, third, etc., most often results in a decreasing quantum efficiency, for each tube, in the order of removal from the system. Investigations are continuing to determine how and where these differences between the methods occur.

#### C. Pinch-off

When the required filling pressure is reached, seal the ion chamber fill tubes.

1. When the pressure in the manifold and ion chamber has reached equilibrium, close the gas fill valve to the ion chamber.
2. Quickly crimp the fill line with a pneumatically operated pinch-off tool, such as shown in Figure 7.
3. With a pair of diagonal cutting pliers (one cutting edge flattened) a second crimp is made 1/16" below the first. Then make a third crimp to separate the ion chamber from the system.
4. Quickly solder the pinch-off end.
5. After soldering a bead at the sealing tip, solder the crimped area to avoid a possible leak. Quickly wipe the soldered area with an alcohol saturated tissue to cool the tubing.

## CALIBRATION

Since a forthcoming paper will give a detailed description of the calibration technique, only a brief mention of the equipment and process used for this will be made at this time.

The equipment (Figure 8) consists of a commercial 1-meter vacuum monochromator,\* a standard hydrogen lamp (GSFC design) which is the light source, a standard ion chamber (Figure 9), a photomultiplier† coated with sodium salicylate, an electronic recording circuit, and an external distillation-vacuum system. A standard ion chamber flange is attached

\*McPherson Monochromator 220.

†RCA 6810A (S11 Photocathode) or EMI 6256, 6256B.

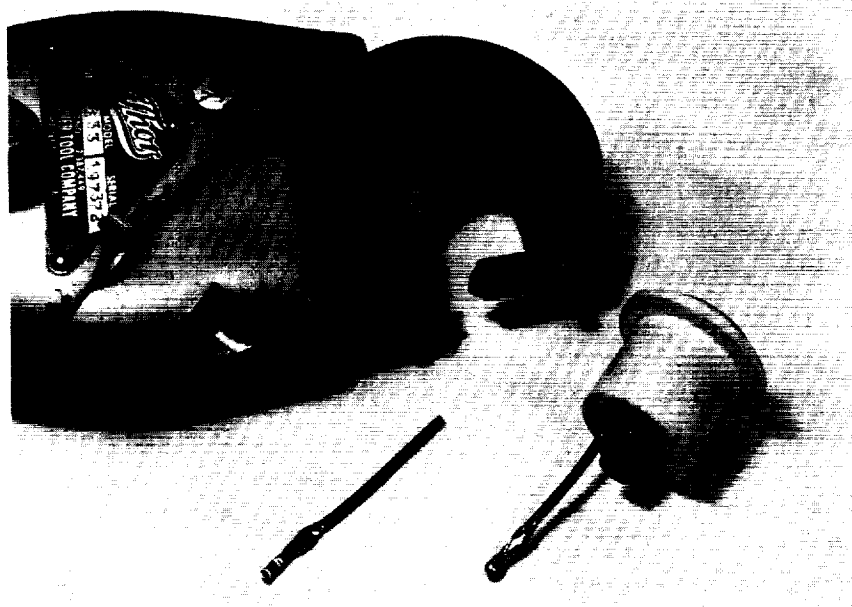


Figure 7 - The pinch-off tool

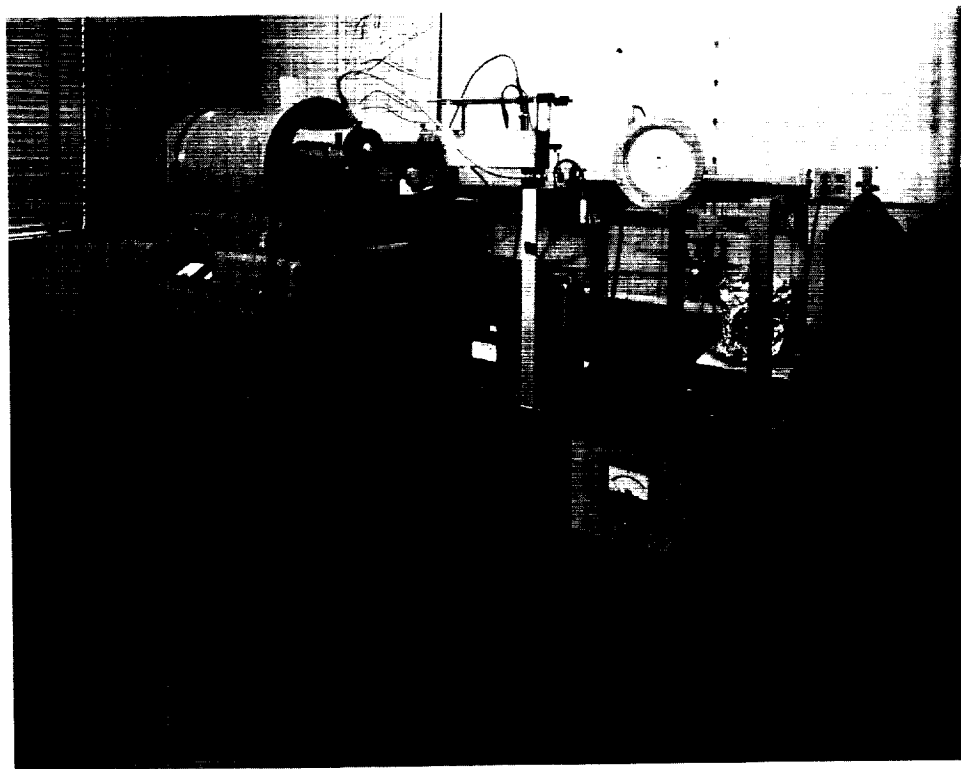


Figure 8— The monochromator (the one pictured is a McPherson 220)

to the exit slit. Since the ion chambers and photomultiplier are frequently interchanged, the exit-slit flange contains a lithium fluoride window to transmit light and to make a vacuum seal with the monochromator vacuum system. This flange contains two "O" rings in order to fit either the standard ion chamber or the ceramic ion chamber (Figures 9 and 10).

#### A. Standard Ion Chamber Calibration

A measure of the absolute photo-ionization yield of an ion chamber can be obtained by utilizing the work of Watanabe (References 4 and 5), who states that the absolute quantum yield of nitric oxide (NO) is 0.81 photons/quantum at Lyman alpha, 1216A. (Reference 2). The standard ion chamber is designed to absorb all of the incident Lyman alpha radiation from the hydrogen-lamp vacuum monochromator source. Eighty-one percent of the photons entering the standard chamber produce an equal number of photoions, which are collected and measured.

Hold the standard ion chamber to the monochromator flange by evacuation through the external distillation-vacuum system. Then back fill with distilled nitric oxide (NO) (usually at 6-8 mm Hg absolute pressure, until a peak signal is obtained at Lyman alpha, 1216A.

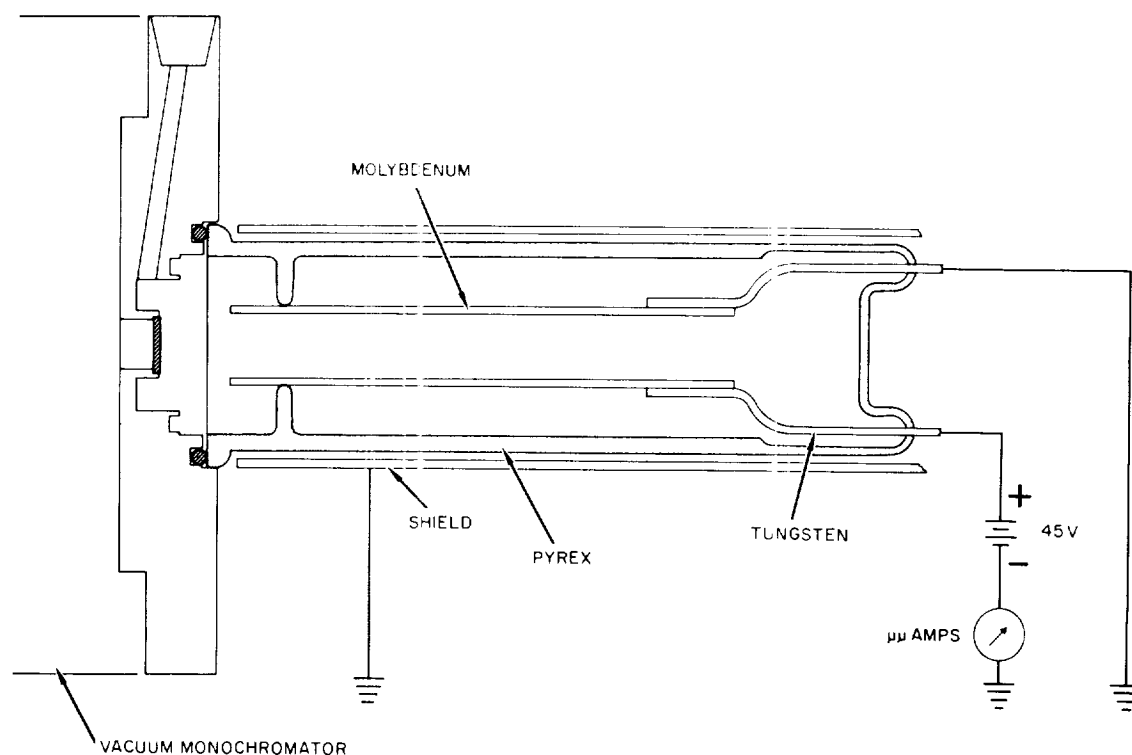


Figure 9 - Standard ion chamber

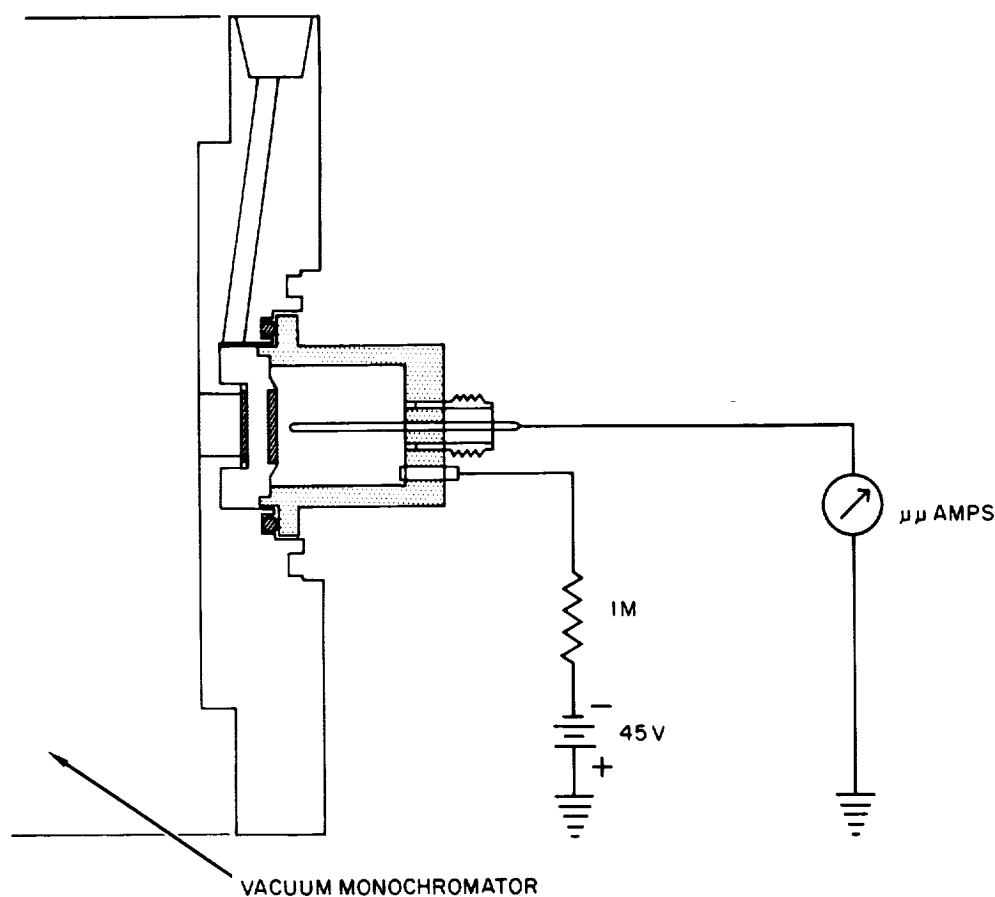


Figure 10 - Ceramic ion chamber

#### B. Ceramic ion chamber calibration

1. Attach the ceramic ion chamber to the monochromator (Figure 10) and subject it to the same hydrogen-source flux as the standard ion chamber. Then make a direct measure of the ceramic chamber's absolute photo-ionization yield at Lyman alpha. This measure can be made as follows:

$$QE_{Ly\alpha} = \frac{\text{Signal from ion chamber at Lyman alpha}}{\text{Signal from standard ion chamber at Lyman alpha}} \times 0.81$$

The quantum efficiency of the ceramic ion chamber is always less than 81 percent since the window absorption, the incomplete gas absorption, and possibly inefficient ion collection reduces the signal.

2. Since many ion chamber-window gas-filling combinations do not respond to Lyman alpha, 1216A, radiation, a method of transferring calibrations from 1216A to some

other specified wavelength is necessary. The flat fluorescence yield of sodium salicylate (Reference 6) provides a convenient method for making this transfer. Sodium salicylate has the optical property of emitting a relatively constant amount of fluorescent radiation for every incident photon in the wavelength region 800-2600A, regardless of the wavelength of the incoming quanta. By exposing a photomultiplier coated with sodium salicylate ( $\text{NaC}_7\text{H}_5\text{O}_3$ ) to the calibrated beam of the monochromator, the flat fluorescent response becomes calibrated at Lyman alpha and can then be used to measure photo-ionization yields at any other wavelength. The quantum efficiency of an ion chamber at a wavelength  $\lambda$  then can be written:

$$\text{QE} = \frac{\text{Ion chamber signal at } \lambda}{\text{NaC}_7\text{H}_5\text{O}_3 \text{ signal at } \lambda} \times \frac{\text{NaC}_7\text{H}_5\text{O}_3 \text{ signal at Ly } \alpha}{\text{Standard chamber signal at Ly } \alpha} \times 0.81$$

### C. Spectral Response Curves

The shape of the ion chamber spectral response curve is determined by a comparison of the ion chamber response to the monochromator beam, at various wavelengths, and the response of the sodium salicylate coated photomultiplier to the same monochromator beam at the same wavelengths. The flatness of the sodium salicylate response gives the information for determining the relative spectral response of the ion chamber at all wavelengths. The absolute quantum yield is then determined in the manner outlined above.

### D. Test Results

Table 1 shows the most important features which have been measured on some of the types of ion chambers constructed at Goddard Space Flight Center. The quantum efficiencies listed represent the variation of the spectral response curves over the peak response region.

Table 1  
NASA Ion Chambers and Measurements

| Gas Filling      | Chemical Formula                   | Window Material | Spectral Response (Angstroms) | QE* (Percent) |
|------------------|------------------------------------|-----------------|-------------------------------|---------------|
| Ethylene Oxide   | $(\text{CH}_2)_2\text{O}$          | LiF             | 1050-1180                     | 10-20         |
| Carbon Disulfide | $\text{CS}_2$                      | LiF             | 1050-1240                     | 50-60         |
| Acetone          | $\text{CH}_3\text{COCH}_3$         | $\text{CaF}_2$  | 1230-1290                     | 8-10          |
| Nitric Oxide     | NO                                 | $\text{CaF}_2$  | 1230-1350                     | 10-30         |
| Nitric Oxide     | NO                                 | LiF             | 1050-1350                     | 10-50         |
| Ethyl Sulfide    | $(\text{C}_2\text{H}_5)_2\text{S}$ | $\text{BaF}_2$  | 1350-1480                     | 10-25         |

\*Based on a value of 81 percent for NO at Lyman alpha.

## ACKNOWLEDGMENTS

Special appreciation is due Mr. Elio V. Serra for suggesting the alumina ion chamber shell and assisting in developing the epoxy sealing technique; Mr. William A. Gallo, Jr. for suggesting the use of the epoxy sealant; and Mr. Francis A. Beall for developing the rotating flange-soldering device and assisting in fabrication of the ion chambers.

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